RESEARCH ARTICLE

Synthesis and Characterization of Magnesium Doped ZnO nanoparticles by Microwave Irradiation Method

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ABSTRACT: Mg doped ZnO nanoparticles were prepared by chemical synthesis associated with microwave irradiation method. The prepared samples were characterised by XRD, FT-IR, SEM, EDAX, UV and PL methods. The X-ray diffraction pattern revealed the prepared sample was crystalline in nature. FT-IR results help to identify the presence of functional groups in the prepared samples. The morphology and purity of samples were analysed by Scanning Electron Microscopy (SEM) and Energy dispersion X-ray Diffraction (EDAX) analysis. The optical properties were studied using ultra-violet spectroscopy (UV) and Photo Luminance spectroscopy (PL). The synthesized materials will be used as catalysis for the degradation of various pollutants, gas sensors, solar cells and LED devices.

KEYWORDS: zinc oxide, magnesium doped zinc Oxide, co-precipitation, Microwave irradiation method, crystalline size

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1. INTRODUCTION

Semiconductor nanocrystals have attracted a great deal of attention from researchers for both their fundamental size dependent optoelectronic properties and their wide range of applications. Zinc oxide (ZnO) is a versatile material that has achieved applications in photo-catalysts, solar cells, chemical sensors, piezoelectric transducers, transparent electrodes electroluminescent devices and ultraviolet laser. Mg-doped ZnO materials have attracted much attention because of their unique UV-luminescent properties based on radioactive recombination of the electron–hole pairs. A peak wavelength of the UV emissions is slightly blue-shifted because of the band gap broadening of were used as received, without further purification [1,2].

The ZnO nanostructures are also being used in several industrial applications such as medicine, gas sensors, etc. The availability of a rich genre of nanostructures makes ZnO as an important material. In this paper, we report the synthesis and characterization of ZnO and Mg doped ZnO nanoparticles by precipitation method and its characterization by using XRD, SEM, UV-Vis, Thermal analysis and Photoluminescence spectroscopic methods[2].Mg doped ZnO dilute magnetic semiconductors prepared using chemical method. Magnetic properties were studied using SQUID technique and morphological and structural properties were studies using SEM and XRD techniques respectively [3,4].

The synthesized of Mg doped zinc oxide nanoparticles were carried out by Microwave irradiation method. ZnO samples were calcined in air atmosphere. The surface morphology of the nanoparticles depends on the calcination temperature and was characterized by scanning electron microscopy (SEM) measurements. Chemical bonding structures in the samples were investigated using a Fourier transform infrared spectroscopy (FT-IR) The effect of the Mg content on the electronic band transitions in the prepared structures were investigated by photoluminescence (PL) spectra at room temperature. In addition, the band gap of the samples was determined by an UV-VIS spectrophotometer at room temperature [5,6].

2. Materials and Methods

2.1 Chemical Synthesis of Magnesium doped ZnO

Magnesium chloride ($MgCl_2$), Zinc Oxide ($C_4H_{10}O_6Z_n$) and sodium hydroxide (NaOH) were received from Merck, Mumbai, India. About 0.32g of Magnesium chloride with 50ml of distilled water was taken in a beaker that was stirred for 30 minutes. Zinc acetate dihydrate 4.9 g were taken in separate beakers with mixture 50 ml of distilled water and stirred for 30 minutes. Then the magnesium chloride solution was

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added into the zinc acetate solution and stirred for 30 minutes. Then the NaOH solution was added drop by drop until the pH reaches to 12. Then the prepared solution was kept for 24 hours at room temperature. The precipitate was washed with demonized water to remove the impurities. Then the collected precipitate was dried for 45 minutes by using microwave oven at 70 watts. The dried cake was grained by using mortar in 40 minutes.

Zinc acetate dehydrate 10.975g was dissolved .with 100ml of demonized water and the solution was stirred for 30 minutes. In this, 10 ml of NaOH solution was added drop wise to above mixture for maintain the pH reaches to 12. The final mixture was stirred for 30 minutes. The gelatinous precipitate was obtained. This obtained precipitate was kept for 24 hours and double washed with demonized water. The precipitate was kept in microwave oven for 30 minutes at 75ºC, and then dries the sample at 400°C around 4 hours. The fine pure ZnO nanoparticles were collected by grained the dried cake using mortar.

2.2 Characterization Techniques

2.2.1 FT-IR

The functional groups of prepared samples were identified using Fourier transform spectroscopy analysis. The spectrum was recorded in the range of 4000-400cm^{-1} region.

2.2.2 XRD

The prepared samples were analyzed using XRD (X-ray Diffraction) technique. XRD pattern predicts the lattice parameters (a&c), unit cell volume and crystalline size of the sample. The XRD pattern of prepared samples was well matched with JCPDS card no: 09- 0432 (hexagonal phase) (7,8). The lattice parameter of the sample was calculated using the following equation:

$$\mathbf{1/d2 = (4(h2+hk+k2)/3a2) +(l2/c2)}$$

Where, d is the spacing between the planes, a and c are the lattice parameter. The unit cell Volume (V) of the sample was described using the given equation:

$$\mathbf{V = (\sqrt{3}/2) + a2+c2}$$

The average crystalline size of the sample was determined by using the scherrer's formula.

$$\mathbf{D = k\lambda/\beta \cos \theta}$$

Where D denotes the average crystalline size of the sample, K represents the broadening constant, **λ** denotes the wavelength of Cu K**λ** radiation source (1.54A°), **β** represents the full width at half maximum, angle of diffraction is denoted by **θ**

2.2.3 SEM and EDAX

The surface morphologies of synthesized ZnO samples were analyzed using Scanning Electron Microscopic analysis (SEM). Energy dispersive spectroscopy is used to identify the elemental composition of the sample.

2.2.4 Ultraviolet-Visible Spectroscopy (UV/Vis)

Molecular (organic) and ionic species capable of absorbing at UV or Visible wavelengths in dilute solutions were analyzed.

2.2.5 Photo Luminescence Spectroscopy (PL)

Photoluminescence spectroscopy is a contactless, flexible, powerful optical method of searching the electronic structure of materials. Light is focused on a sample, the excess energy is absorbed into the material is called photo-excitation (9,10).

3. Results and Discussion

3.1 FT-IR Analysis

The FT-IR spectra identify the molecular composition and chemical bands of materials by producing an infrared absorption spectrum. The FT-IR spectra of pure ZnO and Mg doped ZnO samples recorded in the region of 400−4000 cm−1 are shown in Figure 1. The vibrations of a variety of groups are present at different wave number of IR radiation. The broad peak absorbed at 3930.93cm^{-1} and3861.49cm^{-1} (Alcohol) which be in contact to O-H stretching banded. C-H stretching confirms from the absorption peak of 2802.6 cm^{-1} and 2803.2 cm^{-1} (Alkynes). N=O stretching from the absorption the peaks at 1405.48cm^{-1}(Nitro). Then FT-IR spectrum absorbs the peak at Pure ZnO (ZnO) 3449.87cm^{-1} and Mg doped ZnO (Mg-ZnO) 3445.26cm^{-1} were calculated with the stretching vibrations of N-H (Amine)bond. The present groups were analysis shown in Table 1 and Fig 1.

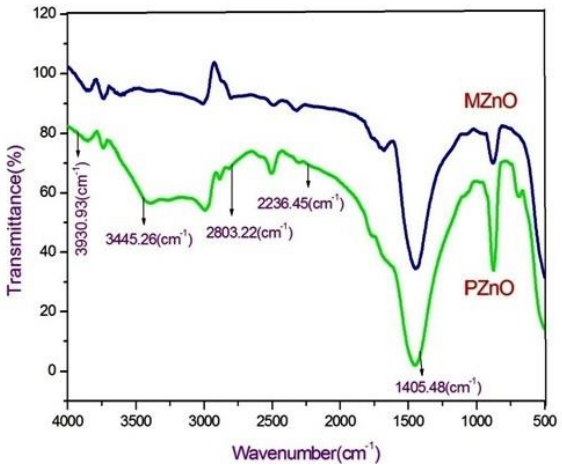

Figure 1: The FT-IR Spectrum analysis of ZnO (pure) and Mg-ZnO (doped)

3.2 XRD Analysis

The XRD pattern of pure ZnO and Mg doped ZnO were shown in Fig: 2.The prepared sample confirms the presence of hexagonal structure and it well matched The broad diffraction peaks of pure ZnO at 2**θ** =36.44°, 63.03°, 68.12° and 69.29° and Mg doped ZnO at 2**θ** = 36.27°,63.03°,68.12°,69.29° are clearly deducted.

The indexed hkl planes are (101), (103), (112) and (201). No impurity peaks were detected in the samples. The average crystalline size (D) of pure ZnO and Mg doped ZnO was 18.67 and 16.98nm. The unit cell volume (V), lattice parameters a & c decreased due to increase in crystalline size and were shown in Table 2.

Table 1: Functional group of Pure ZnO and Mg-ZnO

S.No	Sample Name	Wave Number(cm^{-1})				
		O-H Stretching Vibration(free)	O-H Stretching Vibration(banded)	C-H Stretching vibration	N=O Stretching vibration	N-H Stretching vibration
1	PZnO	2237.43	3930.93	2802.57	1408.04	3449.87
2	MZnO	2236.45	3930.93	2803.22	1405.48	3445.26

Table 2: XRD Analysis of ZnO and Mg-ZnO

Sample Name	2 θ (deg)	FWHM (deg)	D (Å)	Intensity (count)	Crystalline Size(m)	Average Crystalline Size	Hkl	Lattice constant		Lattice Cell volume ($Å^3$)
								a=b	C	
ZnO	36.44 63.03 68.12 69.29	0.47 0.50 0.52 0.48	2.46 1.47 1.37 1.35	119 358 292 163	17.79 18.57 18.43 19.92	18.67	101 103 112 201	3.22	5.19	46.58 47.49 46.12 46.51
Mg-ZnO	36.27 63.03 68.12 69.29	0.50 0.54 0.57 0.53	2.47 1.47 1.37 1.35	816 229 179 89	16.09 16.98 16.73 18.12	16.98	101 103 112 201	3.21	5.21	47.07 45.62 46.78 46.78

3.3 SEM and EDAX

The morphological structure of the prepared nanocomposites was revealed in SEM. The synthesized micrographs of ZnO and Mg-ZnO nanoparticles were crystalline in nature. Fig 3 (a) and (b) shown in the synthesized pure zinc oxide nano particle (ZnO) predicts needle shape structure and Magnesium doped Zinc oxide nanoparticle (Mg-ZnO) spherical shape structure. The particle size of the pure ZnO was 25nm to 103nm in diameter and Mg-ZnO was 71 to 188 nm in diameter.

The EDAX analysis of Pure ZnO and Mg-ZnO are shown in Fig 4. The functional groups of ZnO, Mg and Cl are present in the sample. It clearly confirms the purity of the composition elements. The atomic weight of Zn (40.54%) & O (59.46%) for Pure ZnO. The atomic weight in Pure ZnO was Zn (21.75%), and O (73.70%) respectively. The EDX analysis result is shown in Fig 4.

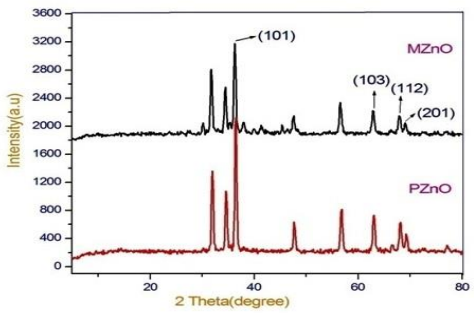

Figure 2: Synthesized sample with XRD pattern for ZnO and Mg-ZnO

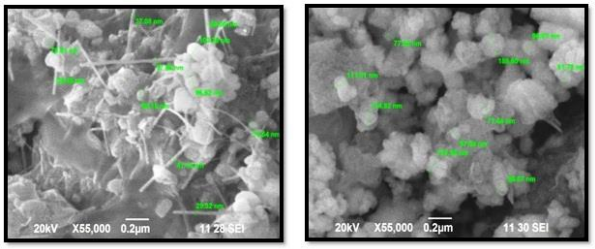

Figure 3: SEM analysis of pure ZnO (a) and Mg-ZnO (b)

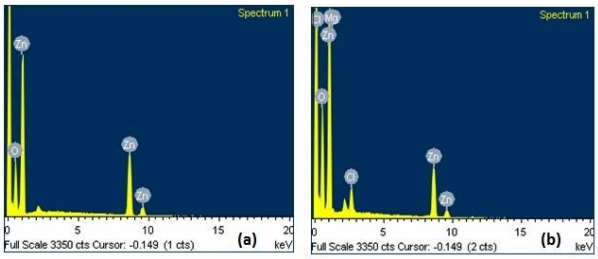

Figure 4: EDAX analysis of ZnO (a) and Mg-ZnO(b)

3.4 UV Analysis

UV-Vis spectroscopy investigates the optical properties and band gap energy of the sample. The absorption spectra of ZnO and Mg-ZnO nanoparticles are found in the wavelength range of 306nm. The band gap energy of ZnO and Mg-ZnO was 4.05eV. Both the sample has same wavelength and bandgap energy due to quantum size effect and electronic structure modifications. There is no change in bandgap energy due to meagre concentration of dopant. Table 5 shows the energy band and wavelength of the particles. The results of UV Spectra of ZnO and Mg-ZnO are shown in Fig 5

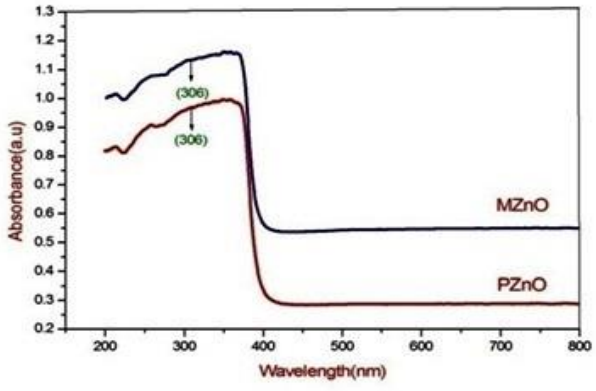

Figure 5: UV-Vis Spectrum of pure ZnO and Mg-ZnO

3.5 PHOTOLUMINESE SPECTROSCOPY

The intensity of emission radiations was absorbed by photoluminescence spectroscopy. The emission radiation of ZnO and Mg-ZnO are shown in Fig 6. The excitation wavelength occurs at the range of 400nm for both ZnO and Mg-ZnO. The emission band of Mg-ZnO located at 440nm. The intensity peaks increased. The standard band gap energy of P-ZnO around the range 3.45 eV to 5.40 eV, while. The calculated band gap energy of both the samples is 4.06 eV, which is in the range of the standard value.

Table 3: Wavelength and Band gap energy of ZnO and Mg-ZnO

S. No	Sample Name	Wavelength (nm)	Band gap Energy (eV)
1	ZnO	306	4.05
2	Mg-ZnO	306	4.05

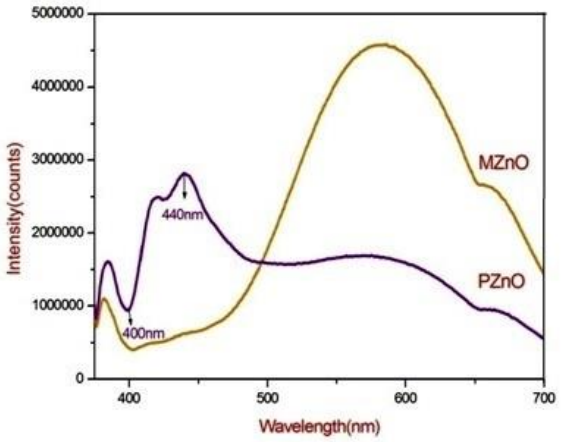

Figure 6: Photoluminescence Spectra of ZnO and Mg-ZnO

4. CONCLUSION

In the present study Mg doped ZnO nano particles were prepared by chemical synthesis associated with microwave irradiation method. The prepared Mg doped ZnO were characterised by XRD, FT-IR, SEM, EDAX, UV and PL. The XRD pattern confirmed the crystalline size of the sample, lattice parameter, and unit cell volume of nanoparticles. The crystalline size increases in Mg-ZnO when compared with pure ZnO. The FT-IR spectrum reveals presence of functional groups. It confirms the Zinc Oxide (ZnO) group present in the sample. The band gap energy and wavelength was determined from UV analysis. The band gap energy of ZnO and Mg-ZnO was 4.05eV. SEM analysis predicts the spherical shaped morphological structure. EDAX analysis confirms the presence of Oxide and Zinc

groups. UV and PL analysis reveals the band gap energy and optical absorption of the sample. The observed Band gap energy was 4.05eV.

REFERENCES

[1] Hammad TM, Salem JK. Synthesis and characterization of Mg-doped ZnO hollow spheres. Journal of Nanoparticle Research. 2011 May 1;13(5):2205-12.

[2] Viswanatha R, Nayaka YA, Vidyasagar CC, Venkatesh TG. 2012. Structural and optical properties of Mg doped ZnO nanoparticles. Journal of Chemical and Pharmaceutical Research. 4(4):1983-9.

[3] Ramesh, P., Rajendran, A., Meenakshisundaram, M. 2014. Green Syntheis of Zinc Oxide Nanoparticles Using Flower Extract. *Cassia Auriculata. J. Nanosci. Nanotechnol.* **2**(1), 41–45.

[4] Cetin SS, Uslu I, Aytimur A, Ozcelik S.2012. Characterization of Mg doped ZnO nanocrystallites prepared via electrospinning. Ceramics International. Jul 1;38(5):4201.

[5] Kalaiselvi V, Mathammal R. 2018. Synthesis and Characterization of Pure and Triethanolamine Capped Hydroxyapatite Nanoparticles and its Antimicrobial and Cytotoxic Activities. Asian Journal of Chemistry. 30(8):1696-700.

[6] Kalaiselvi V, Jayamani N, Revathi M. Comparative Study of Pure Zno and Copper Doped ZnO Nanoparticles Synthesized via Co-precipitation Method. ScieXplore: International Journal of Research in Science, 1(2),73-77,2014.

[7] Kalaiselvi V, Mathammal R, Vidhya N, Surya K. 2019.Synthesis and Characterization of Pure and Capped Hydroxyapatite Nanoparticles. Int. J. Adv. Sci. Eng. 6(1) 1213-1219.

[8] Kalaiselvi, V., Mathammal,R., Vijayakumar,S., Vaseeharan,B., 2018. Microwave assisted green synthesis of Hydroxyapatite nanorods using Moringa oleifera flower extract and its antimicrobial applications. International journal of veterinary science and medicine, 6 (2) 286-295.

[9] Kalaiselvi V, Mathammal R, Anitha P. 2017. Sol-gel mediated synthesis of pure hydroxyapatite at different temperatures and silver substituted hydroxyapatite for biomedical applications. J Biotechnol Biomater. 7(275):2.

[10] Manjunatha, R. L., K. V. Usharani, and D. Naik. 2019. Synthesis and characterization of ZnO nanoparticles: A review. *Journal of Pharmacognosy and Phytochemistry* , 8.3, 1095-1101.

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